THE INITIATION OF YIELDING IN SILICON-IRON**

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SUMMARY

An experimental program was conducted in which the etch pit technique was used for the direct observation of dislocation configurations at various stages of yielding. Poly-crystalline tensile specimens of 3 percent silicon-iron were loaded in tension at constant strain rate and by load pulses.

A new model of the delay-time for yielding at constant applied stress is presented. Three assumptions used are a) no dislocation motion occurs below a critical resolved shear stress, b) the yielding rate is dependent upon the velocity of mobile dislocations, and c) the end of the delay period occurs when yielding of the grains has spread continuously through the thickness of the specimen. This model is consistent with the experimental observations and explains the true static upper yield point and the shape of the strain vs. time curve at constant applied stress. The model also yields reasonable values for the stress concentration factor on grains in the critical cross-section that are least favorably oriented for slip.

I. INTRODUCTION

Several theories for the existence of the upper yield point and associated phenomena have been based on the work of Cottrell (1, 2, 3) and more recently on the work of Johnston and Gilman (4), Johnston (5), and Hahn (6). Many of the reported theories predict the essential features observed during the yielding of body centered cubic (BCC) materials containing interstitial impurities. Yield point drop, delay-time for yielding, pre-yield microstrain, strain ageing, etc., have been described in terms of dislocation locking and the kinetics of dislocation multiplication and mobility. The relative importance of dislocation locking (or unlocking) and of mobility in the various phenomena associated with yielding is not clear.

The early theories failed to account for two important aspects of the problem. First, they did not explain the fact that the yield point depends markedly upon the grain size of polycrystalline materials. Second, it is clear that the release of one dislocation from a Cottrell atmosphere can lead to the generation of many dislocations, as in a slip band, without need for further thermally activated release of pinned dislocations. Experimental evidence for this has been shown by the direct observations of the dislocation structure of slip bands in iron-3% silicon alloy by Low and Guard (7) and Low and Turkalo (8).

Russell, Wood and Clark (9) modified Cottrell's treatment (1) so as to take these two factors into account. They assumed that a certain fraction of all grains must contain slip bands before the onset of macroscopic yielding. The delay-time for yielding according to this theory, is the mean time for the thermally activated release of a dislocation within a grain from its Cottrell atmosphere. The time for dislocations to move across a grain, cross-slip, generate and form a slip band was assumed to be negligible compared with the mean time for dislocation release. Reasonable agreement with experimental measurements of the influence of stress, temperature, and grain size upon the delay-time for yielding was obtained by suitable adjustment of the undetermined constants in this theory.

An alternative hypothesis is that the delay-time may be governed by the kinetics of dislocation multiplication and motion as suggested by Johnston and Gilman (4) and Johnston (5). Hahn (6) and later Cottrell (3) have applied dislocation kinetics theories to the yielding of polycrystalline materials. Hahn obtained the correct stress and temperature dependence for the delay-time for yielding. However, Hahn's treatment does not consider the heterogeneous nature of polycrystalline materials. It does not explain the disappearance of the yield drop with increasing grain size, does not correctly describe the observed pre-yield microstrain and does not predict a true static upper yield point.

Dislocation velocity in individual grains of polycrystalline silicon-iron has been measured by Moon and Vreeland (10). The yield delay time of polycrystalline silicon-iron was measured in the present investigation. These measurements permit a quantitative comparison of the calculated delay times based on the observed dislocation kinetics, and the experimentally measured delay time.

II. MATERIAL AND TEST SPECIMENS

The material used in this investigation was the same as that used in (10). Polycrystalline specimens were prepared by cold rolling and recrystallization as indicated in Table A.

TABLE A

Polycrystalline Specimens

Cold Reduction (%)	50
Recrystallization Temperature (F°)	1290
Recrystallization Time (min.)	60
Mean Grain Diameter (in.)	0.001
Dislocation Density (cm^{-2})	10^6 - 10^7

III. EXPERIMENTAL

Etching Technique

The methods used in (10) were employed in this investigation to reveal dislocation intersections with the observation surface. The configuration of dislocations within the interior of specimens was observed by preparing sections through the specimen in that portion of the gage length in which strain was measured (see below).

Mechanical Testing

Two basic types of test were conducted on the various specimens used in this investigation utilizing the equipment described in (10). First, constant strain-rate tests were conducted to determine the static stress-strain curves for polycrystalline specimens. Second, rapidly applied constant stress pulse tests were conducted to determine delay-times for yielding in fine grain polycrystalline specimens. The durations of the stress pulses were adjusted so as to remove the load either at various stages during the delay time or after yielding had occurred. All tests were conducted at room temperature of about 75°F. The strain in the specimen was detected by means of two foil type strain gages mounted on opposite sides of the gage section. During some tests the total strain was recorded while during other tests only the plastic strain in the specimen was recorded. The maximum sensitivity of the strain measurements was 20×10^{-6} in,/in. per inch of trace deflection on the oscillograph recording paper employed in rapid load tests.

INSERT FIGURE 1

A reproduction of a typical oscillograph record of the applied load and the strain vs. time is shown in figure 1. The delay-time for the initiation of yielding as shown for specimen M21 was 30×10^{-3} sec. The pre-yield plastic microstrain at the end of the delay-time was 140×10^{-6}. The delay-time as shown terminates with ~~represents~~ the initiation of the very rapid increase in strain rate which precedes the development of a Lüder's band. The high sensitivity of the strain measuring device reveals the continuous nature of the growth of an embryonic Lüder's band when the strain detected is the average value on a surface area 3/16 by 3/16 in. (i.e., gage dimensions). Lüder's strain had not occurred under a significant fraction of the gage when the strain trace reached the edge of the recording paper. The load drop recorded for specimen M21 indicates that the strain rate of the specimen increased until the Rapid Load Testing Machine was no longer capable of maintaining the applied load at the required extension rate.

A. ~~Static and~~ Constant Strain Rate Tests

~~Static tests and~~ Constant strain-rate tests were made with a 10,000 pound Instron tensile testing machine. All test data ~~for static tests and for constant strain-rate tests~~ were recorded on the X-Y chart recorder on the Instron.

Constant strain rate tests were conducted to determine the plastic strain vs. stress curves for fine grain polycrystalline specimens. One group of specimens was loaded to a point just below the upper yield point and then unloaded to permit determination of the residual pre-yield microstrain. A second group of specimens was strained beyond the upper yield point.

A total of nine fine grain polycrystalline specimens was strained in the above manner to determine the early portions of the static stress-strain relation with highest possible sensitivity. Seven specimens were strained at 0.010 in./min. crosshead speed and two were strained at 0.020 in./min. crosshead speed. Five of the specimens were strained beyond the upper yield point. The results are given in Table B.

TABLE B

Results of Constant Strain Rate Tests in Polycrystalline Specimens

a. Specimens Strained Below Upper Yield Point

Specimen No.	Applied Stress $lb/in.^2$	Crosshead Rate in./min.	Pre-yield Microstrain 10^{-6}
M28	75,100	0.01	44
M34	70,100	0.01	64
M36	71,500	0.01	118
M38	71,300	0.01	68

b. Specimens Strained Above Upper Yield Point

Specimen No.	Upper Yield Stress lb/in.^{2}	Lower Yield Stress lb/in.^{2}	Crosshead Rate in./min.	Pre-yield Microstrain 10^{-6}
M 3	74,200	68,500	0.01	134
M 5	76,600	70,200	0.01	97
M11	73,700	68,500	0.02	--
M12	71,600	67,100	0.02	--
M27	73,000	---	0.01	280

B. Pulse Loading Tests

Pulse loading tests were made on ten polycrystalline specimens to ascertain if this material exhibited a delay-time for the initiation of macroscopic yielding similar to that found for annealed low-carbon steel as reported by Clark and Wood (11). These tests were conducted at stresses ranging from 71,500 lb/in.^{2} to 75,500 lb/in.^{2}. Surfaces and cross-sections of some of these specimens were etched to reveal the dislocation configurations associated with various stages of development of the pre-yield plastic microstrain. Table C contains the results of the delay-time tests.

TABLE C

Results of Delay-Time Tests in

Polycrystalline Specimens

Specimen No.	Applied Stress $lb/in.^2$	Delay-Time sec	Pre-Yield Microstrain 10^{-6}
M15	75,500	>0.050	168
M17	72,300	0.050	112
M21	73,300	0.030	140
M22	72,400	0.015	158
M23	72,500	>0.050	153
M24	71,500	0.010	70
M39	74,600	0.170	--
M40	73,600	0.250	267
M43	72,700	0.263	165

The pre-yield microstrain behavior during the delay-time period was similar to that observed in low carbon steel by Vreeland, et. al. (12). The relaxation of the highly stressed favorably oriented grains causes an initial high strain rate which is reduced as more of the load is shifted to grains with higher compliance and less favorable orientation for slip. As these grains begin to yield, the strain rate increases (provided the applied stress is above the upper yield stress) and the catastrophic yield process leading to the formation of a Lüder's band is begun.

The complete relaxation of a grain does not occur with the formation of the first slip band. A family of slip bands is observed to build up within the grain by the addition of one new slip band at a time adjacent to an existing slip band at a characteristic distance h. The characteristic spacing, h, must be a function of the stress and test temperature, but does not appear to be a very sensitive function of stress. A value of $h = 0.91 \times 10^{-4}$ in. has been reported for statically loaded silicon-iron by Hibbard and Dunn (13). Average values for h in randomly oriented grains in this investigation were 0.95×10^{-4} in. for static tests and 0.96×10^{-4} in. for pulse tests.

IV. DISCUSSION

Delay-Time for Yielding in Polycrystalline Specimens

The delay-time for yielding in fine grain silicon-iron observed in this investigation ranged from 0.010 to 0.263 sec for applied constant stresses between 71,500 and 74,600 lb/in^2. Assuming an applied stress of 72,400 lb/in.^{2} and an ideally oriented grain (Schmid Factor = 1/2) within a polycrystalline matrix, the nominal resolved shear stress on the grain would be 36,200 lb/in^2. The velocity of a dislocation in this grain would be approximately 340 cm/sec. (10). At this velocity the dislocation would cross the ideally oriented grain in about 7.5×10^{-6} sec. The delay-time for yielding is at least a factor of 10^3 to 10^4 greater than the time necessary for individual dislocations to traverse the ideally oriented grains in a specimen.

Grain boundaries are the primary source for slip band nucleation in annealed material, and a critical resolved shear stress greater than the friction stress is required to initiate slip in a fully annealed grain (10).

These observations will be used in the delay-time model to be presented below. The sequence of events leading to the formation of a Lüder's band has been observed in this investigation by the use of the pulse loading and electrolytic etching techniques. These observations, together with the observations of the stress dependence of dislocation velocity will now be used to develop a semi-quantitative description of the dislocation mechanics leading to the formation of the Lüder's band.

The following explanation of the yielding of silicon-iron is based on three assumptions: first, that dislocation sources are not activated until a critical resolved shear stress τ_c is reached which is greater than the friction stress τ_f; second, that the rate of yielding is governed by the dislocation velocity which is a sensitive function of resolved shear stress (the resolved stress varies from grain to grain); and third, that the delay-time is essentially given by the time for the yielded region to spread continuously through the thickness at some section of the specimen. This section is called the critical section.

The strain vs. time relation for fine grain polycrystalline silicon-iron subjected to a constant stress can be explained as follows. The initial relatively high plastic strain rate is the result of the relatively high dislocation velocity in the grains with the highest resolved shear stresses. A variation in resolved shear stress from grain to grain is the result of two factors: a) the grains are oriented differently and hence have different Schmid factors, and b) the elastic moduli of neighboring grains differ when referred to common axes because of elastic anisotropy (14). The strain rate decreases as the highly stressed grains are relaxed and the fraction of the total grains relaxed at any time will be dependent on the applied stress. Applied stresses less than the static upper yield point and great enough to produce τ_c in some grains produce a strain rate that decreases to zero. The structure at this point shows essentially isolated relaxed grains because stresses on adjacent grains are insufficient to generate slip bands. As the stress is increased, more grains relax and clusters of relaxed grains are formed. An applied stress equal to the static upper yield stress will result in a zero strain rate following the high initial rate. However, the duration of this condition is indeterminate. The effect of the relaxation of those grains which have yielded prior to general yielding is to increase the resolved shear stress on less favorably oriented grains to a value which will initiate slip bands in the adjacent grains. The clusters of relaxed grains cause a local stress concentration on adjacent grains (proportional to the square root of the cluster diameter). When the upper yield stress is reached, the stress concentration is sufficient to cause one of the clusters of yielded grains to extend through the thickness of the specimen. At this stage, the stress concentration factor increases significantly and the band rapidly spreads to cover a cross-section of the specimen. When a cross-section of the specimen is completely covered by relaxed grains the Lüder's band is distinguishable by its characteristic surface deformation and propagates as a stable front along the length of the specimen. The propagation velocity of the Lüder's band is less than the growth rate of the embryonic band across a cross-section because the value of the stress concentration factor decreases when the elastic-plastic transition region extends across the entire specimen. The time for yielding to occur under constant applied stress is then essentially the time for an embryonic Lüder's band to grow across the thickness of a specimen.

The delay-time required to relax a grain by the observed mechanism, i.e., one slip line at a time, would be about d/h times the traverse time necessary for the formation of one slip band across the grain (d = grain diameter and h = slip band spacing). The average relaxation time for an ideally oriented grain at an applied stress of 72,400 lb/in^2 would be ten times the traverse time for a single slip line, or about 7.5×10^{-5} sec, if the stress in the grain is assumed to be constant. This relaxation time is still 10^2 to 10^3 less than the observed delay-time. However, all grains are not ideally oriented and the time to relax less favorably oriented grains is significantly longer. The fine grain specimens contained approximately ten grains through their thickness, and the delay-time for yielding can be calculated from the dislocation velocity vs. stress data if the stress is estimated for each grain at the critical cross-section. Such an estimation is very difficult. In view of the very high sensitivity of dislocation velocity to stress, the delay-time will essentially be governed by the time to relax the grain that has the lowest stress (i.e., lowest Schmid factor). Therefore, the delay-time will be approximately given by

$$t_D = \frac{d^2}{h\, V_{min}} \qquad (1)$$

where d is the diameter of the grain in the critical section having the lowest resolved shear stress, h is the spacing between adjacent slip bands in the grain and V_{min} is the dislocation velocity in the grain.

The minimum velocity V_{min} is given by

$$V_{min} = (\tau_{min}/\tau_o)^n \tag{2}$$

but again τ_{min} cannot be accurately estimated. The expression

$$\tau_{min} = m\ S\ \sigma \tag{3}$$

where

m = stress concentration factor due to surrounding relaxed grains and anisotropy,

S = Schmid factor and

σ = nominal applied tensile stress,

will be used to calculate a value for τ_{min} based on the observed values of t_D, h, $\overline{d}$, n and τ_o obtained in this investigation.

Combining the above expressions gives the delay-time as

$$t_D = \left(\frac{\tau_o}{mS}\right)^n \frac{d^2}{h}\,\sigma^{-n}\ . \tag{4}$$

Consider a typical delay-time test shown in Table C. For specimen M21, t_D = 0.030 sec, h = 10^{-4} in., $\overline{d}$ = 10^{-3} in., n = 32.6 and τ_o = 31,700 when the nominal stress was 73,300 lb/in.^{2} . From equation (1),

$$V_{min} = \frac{1}{3.0}\ \text{cm/sec}$$

and from equation (2),

$$\tau_{min} = 30{,}650 \text{ lb/in.}^2$$

which, when substituted into equation (3), gives

$$mS = 0.418$$

The value of 0.418 is reasonable for the product of stress concentration factor and Schmid factor for a poorly oriented grain, in view of the fact that S can be as low as 0.278.

The stress dependence of the delay-time is the same as the stress dependence of the dislocation velocity. The temperature dependence of the delay-time results from the temperature dependence of the dislocation velocity as postulated by Hahn (6) and Cottrell (3).

The foregoing discussion has explained the appearance of a true static upper yield point, the observed plastic strain vs. time behavior at constant stress and the delay-time for yielding at constant stress in polycrystalline silicon-iron. Additional tests are required to test the predictions fo the dependence of delay-time on grain diameter.

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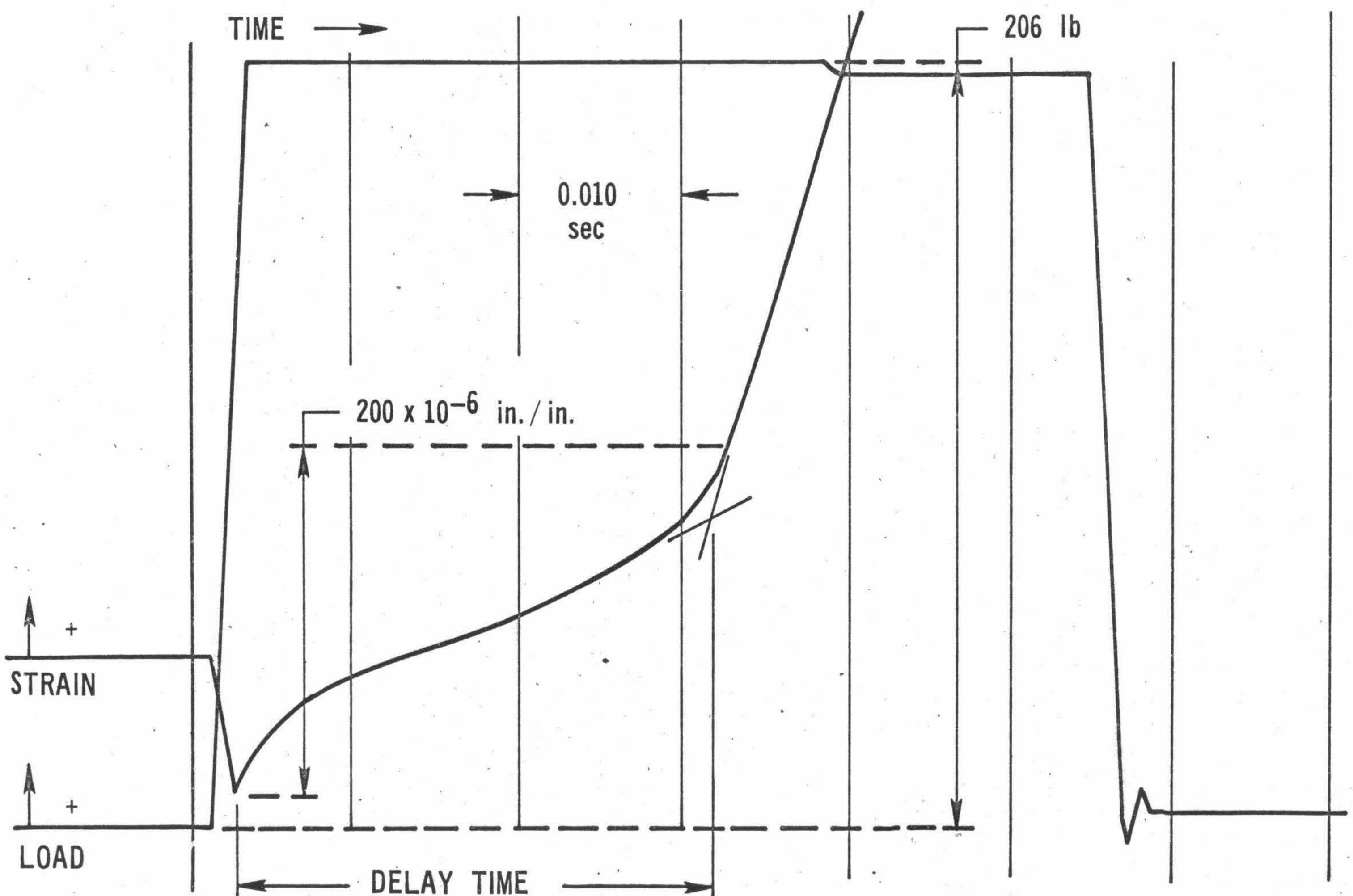

FIGURE TITLE

Fig. 1. Reproduction of pulse loading test record for specimen M21.